Fermi National Accelerator Laboratory
PO Box 500
Batavia, IL 60510
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Report on Batch 1 of Polycrystalline Niobium for ILC Cavities to be Fabricated at AES

A. Aizaz[2], C. Antoine[1], M. Battistoni[1], P. Bauer[1], T. Bieler[2], C. Boffo[1], D. Burk[1], C. Cooper[1], M. Foley[1], T. Grimm[2], D. Hicks[1], H. Jiang[2], O. Lira[1]

1 - Fermilab – Technical Division
2 – Michigan State University

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1) Introduction

Fermilab recently received a first batch of niobium for the fabrication of ~10 1.3 GHz 9-cell cavity prototypes for the ILC project R&D. The batch consisted of 244 square sheets, 10.4" wide, from Wah-Chang ATI. The material is of the polycrystalline, rolled type. The results of visual inspection and Eddy current scanning were reported in other internal notes (TD-05-50 and TD-06-010). This document reports on non eddy-current related investigations of this material. In particular we discuss the measurements conducted to investigate the non-isotropic forming properties that were encountered during the forming of the first six half-cells at AES. These findings together with unusual "stripes" found in the Eddy current scanning triggered an extensive characterization of the material, including micro-structure studies and stress-strain measurements at Michigan State University. The results of this investigation will be reported here.

2) Initial Findings

As illustrated in the example in Figure 1 the first batch of six half-cells hydro-formed at AES[1] are not round at the equator as they should be (specified max error: 0.1 mm). The deviation from the nominal diameter is up to ±0.25 mm (~0.1% on a ~23 cm diameter), such that the equator footprint was strongly elliptical. One should also note that this result was obtained after 6 consecutive forming steps (rather than the usual 1-2). AES informed Fermilab about the difficulties with the forming and the observation of non-isotropic behavior of the material during forming. In one case it appeared like there was one area of a blank that was significantly harder then the surrounding material.

The Eddy current scanning of these niobium sheets also revealed a new feature, "stripes" running parallel to the sheet edges (and thus presumably parallel or perpendicular to the rolling direction), such as shown in Figure 2. These stripes have since been shown to be topological features, i.e. a surface ripple with peak-to-peak amplitudes of ~4 μm and spaced ("wave-length") by a little less than 1". Figure 3 shows the result of a profilometric stylus measurement across and along these features on sheet 137, clearly showing the small ripple in the "across" case on top of a longer range height variation,

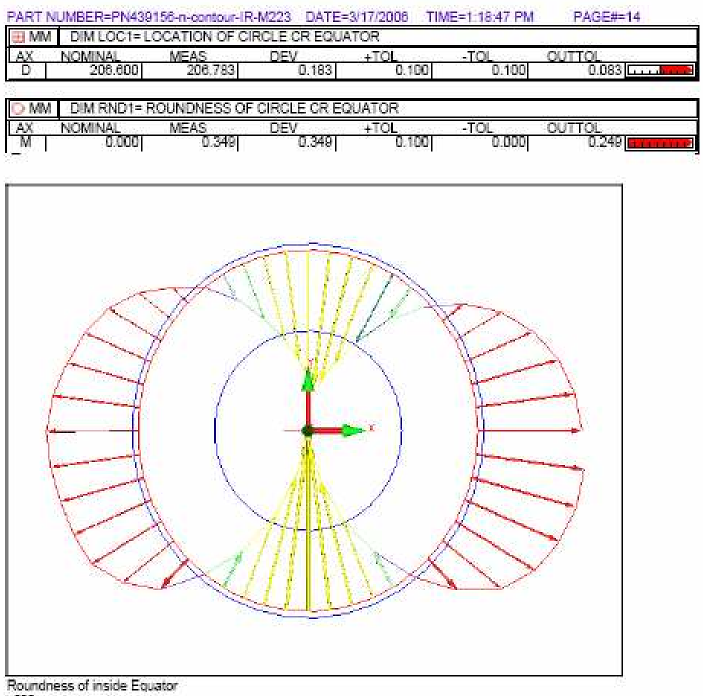


Figure 1: Result of geometrical measurement of equator of one of the initial half-cells formed from the batch 1 Wah-Chang sheet material. The deviation from the specified, circular shape are exaggerated in this plot (Courtesy of R. Riley - Fermilab).

[1] Advanced Energy Systems Inc, Medford, NY, http://www.aesys.net

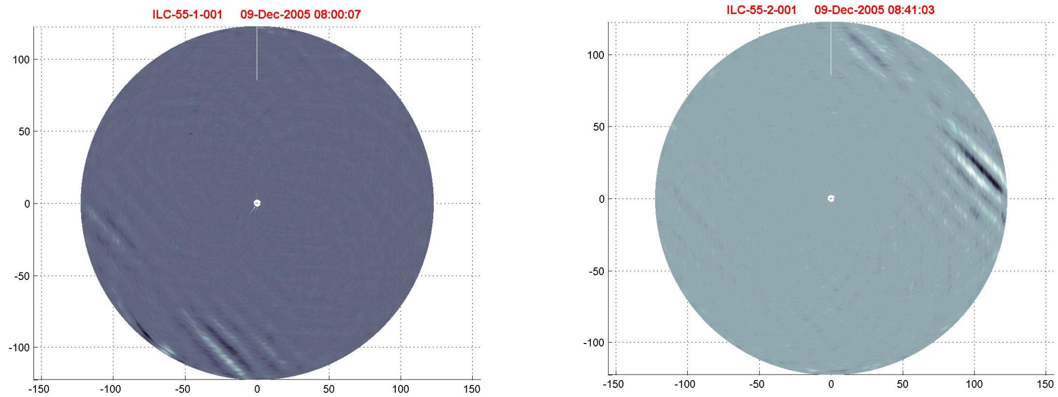


Figure 2: Results of eddy-current scan

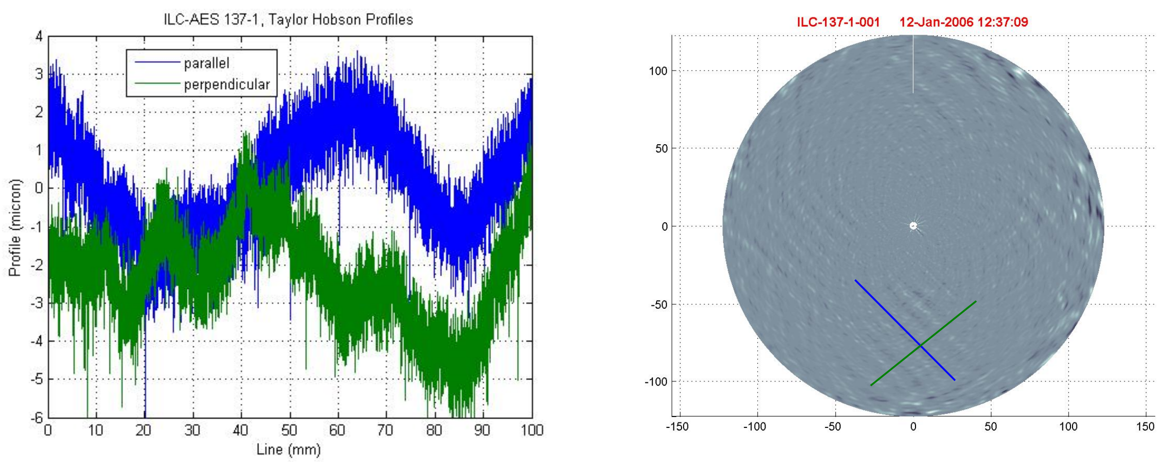


Figure 3: Profile parallel and perpendicular to "stripes" in scans on sheet 137.

which exists in both directions. This issue is discussed further in TD-06-010. Note that all these height variations do not violate the specification for the rolled sheets and are expected to be benign from the cavity performance point of view. Having uncovered the origin of the "stripes", we are now uncertain about their relation to the forming problem.

According to J. Belfanti (product engineer at WahChang) the material was produced according to a standard process, which includes three alternating reduction and heat-treatment steps. The cross-rolling reduces the sheet thickness by 80% (40% in 2 perpendicular directions) and the intermittent heat treatments were performed at approximately 850°C, 800°C and 700°C (always 2 hrs).

To better understand the issues at hand we launched an extensive material characterization program, including mechanical testing and texture measurements at MSU as well as a heat treatment study at Fermilab. The heat treatment study consisted in

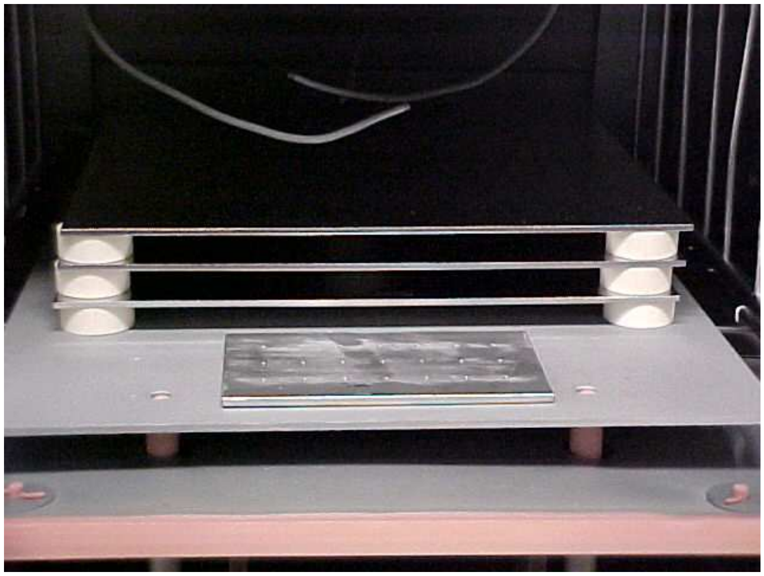


Figure 4: ILC sheets in small vacuum furnace.

heat treating several sheets in the IB4 vacuum furnace[2] for 3 hrs at 750°C (Figure 4), as suggested by C. Antoine. The results of these measurements will be reported in the following.

2) Microstructure Measurements

To measure precisely grain size and microstructure Orientation Imaging Measurements (OIM) were performed at MSU on the samples provided by Fermilab. The samples, 1cm^2 squares, were cut from sheets with electro-erosion. The cut surfaces of interest were then mechanically polished and placed in the special SEM with orientation imaging capability at MSU. The OIMs were performed by H. Jiang. Two measurements were performed for each sample, one for each of two perpendicular thin edges (=cut surfaces) of the sample. In this way two perpendicular projections of the grain shapes across the sheet thickness were obtained.

Two texture samples were cut from sheet 217 to represent the before heat treatment condition. The texture in the before heat treatment case is shown in Figure 7. Two more texture samples were cut from an area partially within one of the "surface waves" (disc 96) to further elucidate their origin. Figure 6 shows the area from where the samples were cut, together with the resulting texture across the sheet thickness. Two texture sample were cut from sheet 55 after the 750°C/3hrs heat treatment. The texture in the after heat treatment case is shown in Figure 8.

[2] Thanks to Tug Arkan / Fermilab for helping us out with the heat treatment.

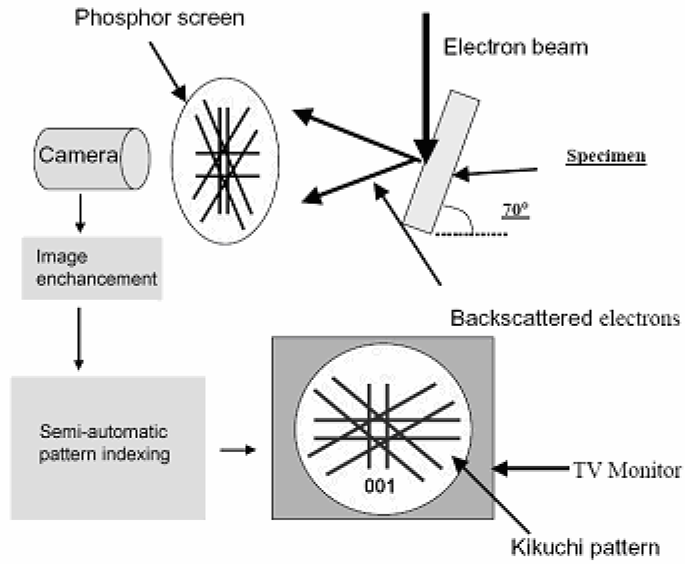
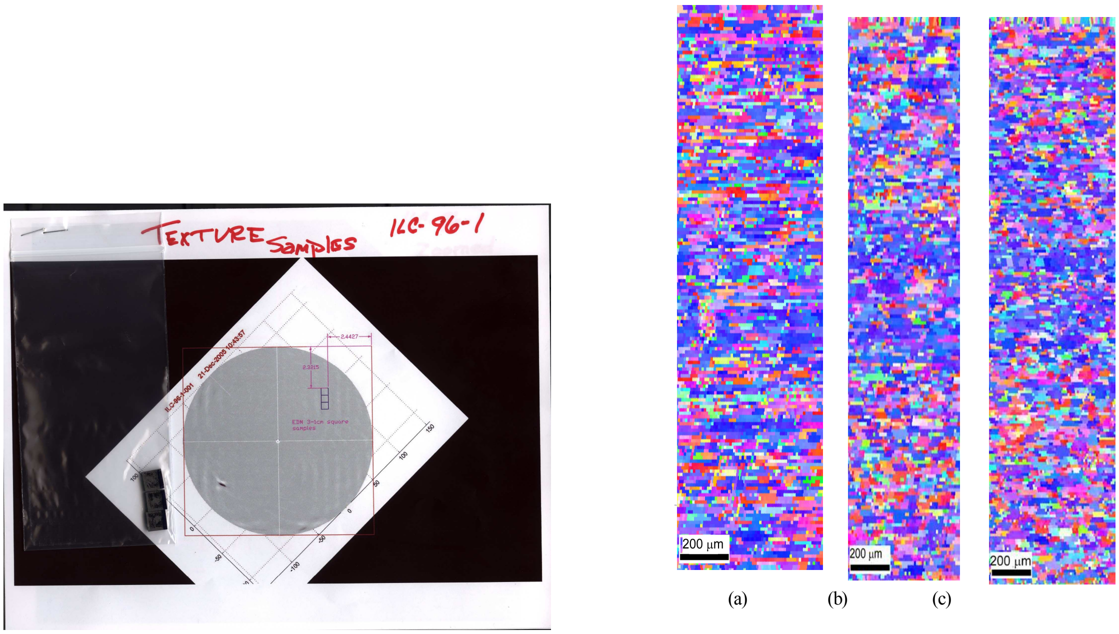


Figure 5: Schematic of Orientation Imaging Measurements.

The texture analysis in the before heat treatment condition shows that the material is only partially re-crystallized with grains being elongated (aspect ration around 2:1). The case in Figure 6, showing the texture in two cross-sections that are perpendicular to each other, indicates that the elongation is more or less similar in both rolling directions. The grains thus are "pancake-shaped" (flattened along sheet normal, elongated in sheet plane). Furthermore, as clearly visible in Figure 7, there are also some bands of larger grains, differing from the finer grains in the rest of the cross-section. This is a worrisome feature in what refers to the forming characteristics of the material. The texture inhomogeneity through the thickness is pronounced in the before heat treatment samples. The grain orientation is not completely random. The 111 orientation is preferred for rolling. There is also more than desired texture in the 001 orientation, which is not ideal for forming.

The texture after the heat treatment is clearly different: grains are now larger and more equi-axed. Also the texture has changed, with the 111 peak being more pronounced and the 001 texture weakened.

Figure 9 shows the grain size distributions in several samples (before and after heat treatment). The grain sizes measured before heat treatment correspond rather to ASTM 6&7 than the specified ASTM 4-5. The grain sizes after heat treatment are therefore (and luckily) close to those specified.



(a) (b) (c)

Figure 6: texture samples cut from areas giving a "stripe" in the Eddy current scan. Samples (a) inside the stripe (b) outside the stripe (c) perpendicular (across) to stripe

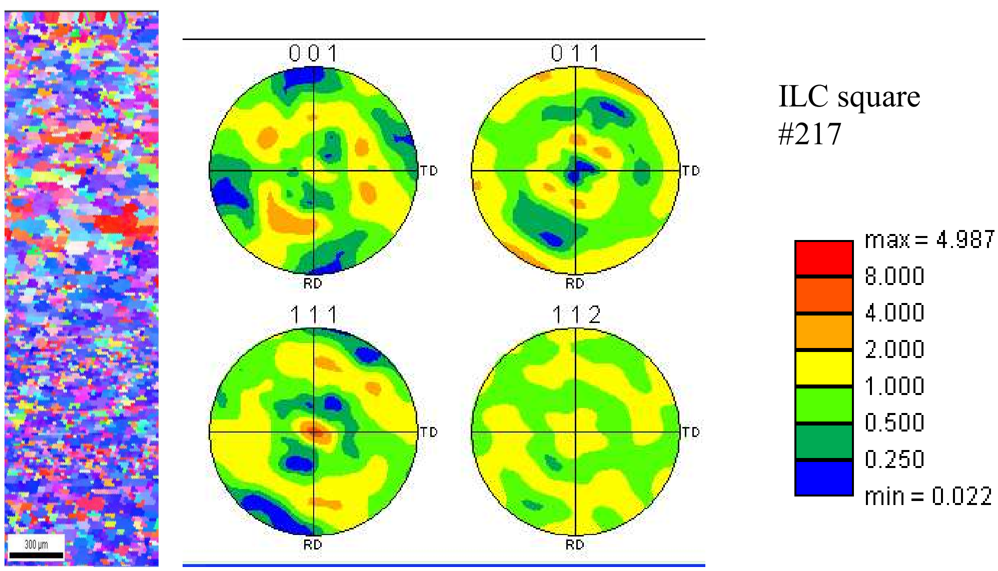


Figure 7: Texture measurements before (left, sheet 217) heat treatment. 111 peak is small and 001 orientation peaks are larger than desired.

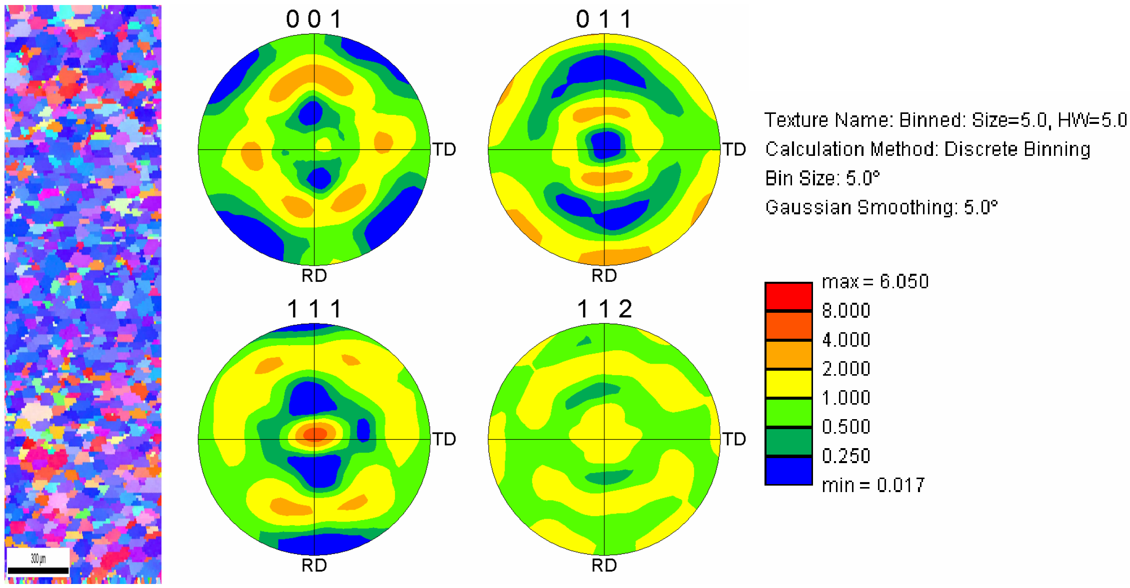


Figure 8: Texture measurements after (right, sheet 55) heat treatment. Pole figure indicates reduced texture in 001 and increased 111 orientation.

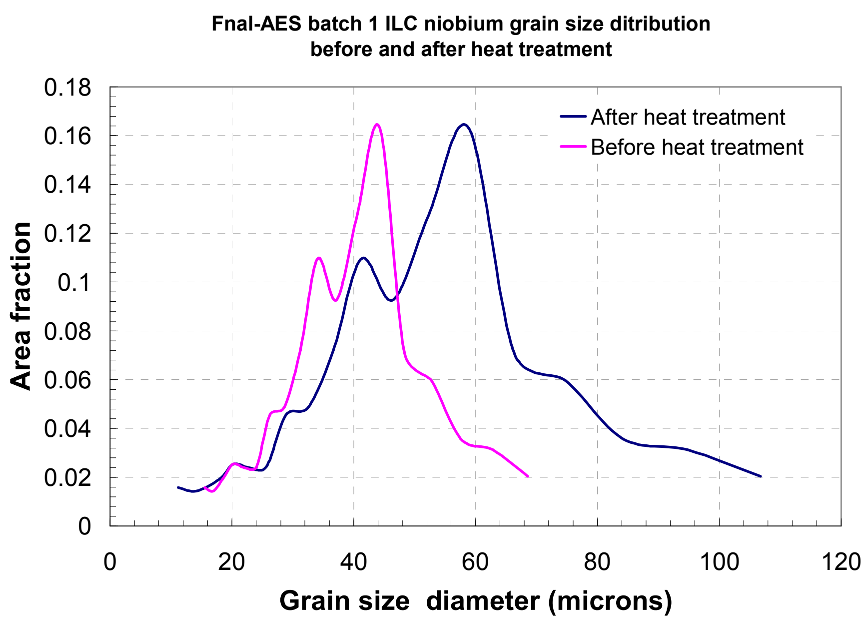


Figure 9: Grain size before and after heat treatment.

The change in microstructure from the before to the after heat treatment condition is also clearly revealed in the following metallo-graphical analysis performed at Fermilab. Sections across the sheet thickness in two perpendicular orientations (parallel to the two sheet edges) are shown in Figure 10. As with the samples sent to MSU for OIM the before heat treatment sample was cut from sheet 217. The after heat treatment sample was cut from sheet 55. The first picture for the before heat treatment case shows horizontally elongated grains. After the heat treatment the grains are larger and more equi-axed, with no more elongated grains along any of the two perpendicular directions sampled. Figure 11 gives a higher resolution micro-graph of one of the samples after heat treatment.

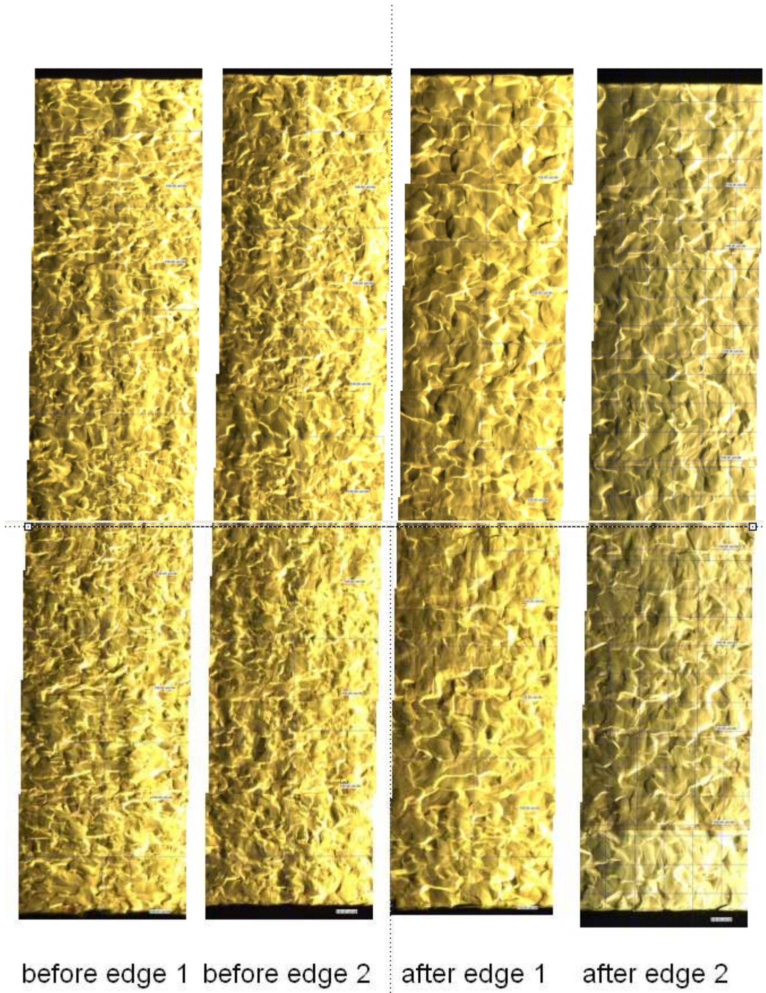


Figure 10: Micrographs across the sheets parallel (left, edge 1) and perpendicular (right, edge 2) of before (from sheet 217, left two picture) and after (from sheet 55, right two pictures) the heat treatment (similar scales).

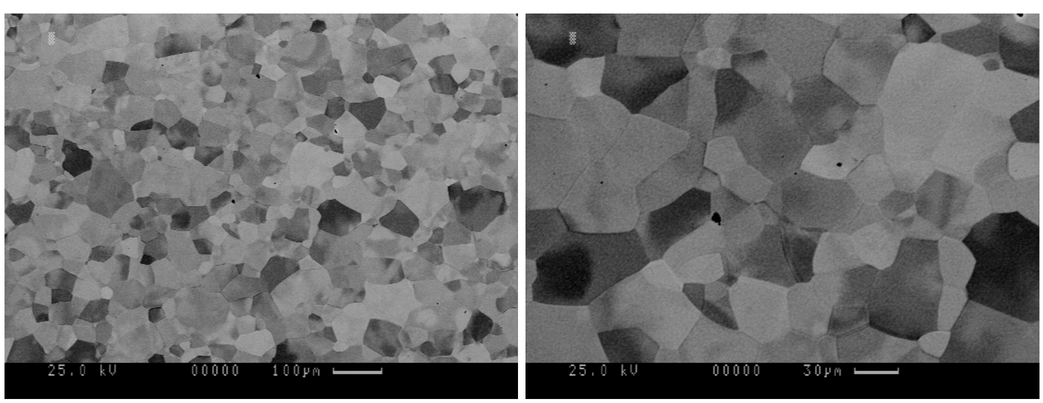


Figure 11: MSU micrographs of one texture sample after heat treatment.

3) Mechanical Measurements

A strong indicator of micro-structural issues is the mechanical stress-strain characteristic, especially if taken along different directions. Such measurements can reveal material texture and mechanical anisotropy as well as predict forming behavior. Stress-strain measurements at room temperature were performed at MSU by H. Jiang on samples provided by Fermilab. Samples representative of the before and after heat-treatment condition were supplied. As with the texture samples they were cut from sheets with an electro-erosion cutting machine. Figure 12 shows the samples after cutting. The samples represent different directions with respect to the sheet edges, one of which is presumably parallel to the rolling direction.

The tensile tests were performed on a 4200 series Instron testing machine, model number 43K2. The set of samples were machined out of the parent sheets, at 0°, 25°, 65° and 90° into the standard "dog bone" shape, with a gage length of 30 mm, and gage width of 6.354 mm and thickness 3.218 mm. The strain rate is 3×10^{-3}/s. The results are shown in Figure 13 and Figure 14.

Before the heat treatment the yield strength varied by up to 22% between the different directions. Most importantly for the formability of the material, however, the strain hardening coefficient varied by 30% between the different directions. That maybe caused by the inhomogeneous grain size and non-ideal (001) texture of the material. After the heat treatment the yield strength dropped by ~20 MPa, an expected softening of the material following further re-crystallization. Although the 25°direction gave a yield strength that was higher than that of the others, the strain hardening coefficient now varies much less (Table 1). As expected from the reduction in yield strength the strain-hardening coefficient has also increased. This corroborates the findings from the microstructure analysis and indicates that the formability of the material should have been improved by the heat treatment. The mechanical measurements, however, also indicate that some of the mechanical anisotropy is still present in the material.

Figure 12: Samples for stress-strain measurements in the before (left, cut from sheet 217) and after (right, cut from sheet 55) heat treatment condition.

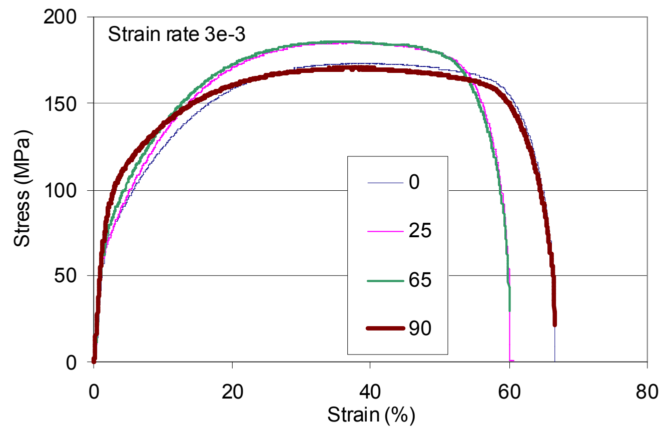


Figure 13: Stress-strain measurements before the heat treatment conducted at room-temperature (sheet 217) at MSU (Courtesy of H. Jiang, T. Bieler). Samples were cut at different angles to the sheet edge (see Figure 12).

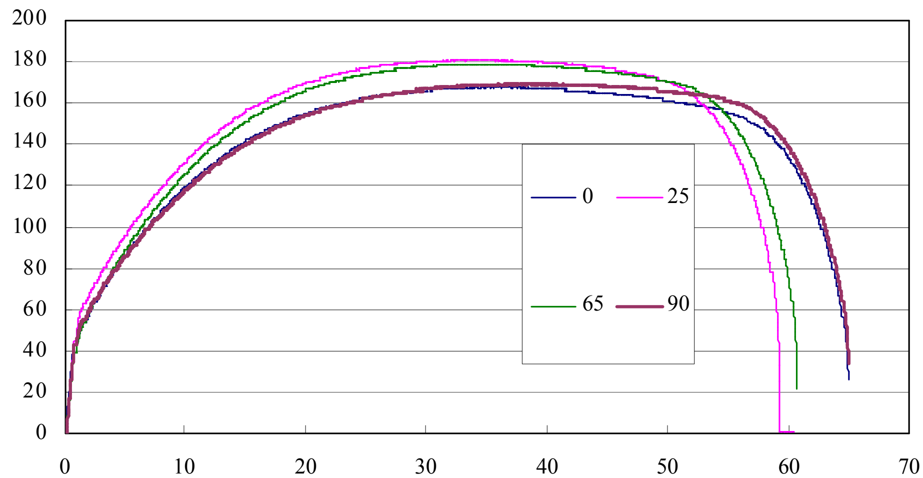


Figure 14: Stress-strain measurements after the heat treatment conducted at room-temperature (sheet 55) at MSU (Courtesy of H. Jiang, T. Bieler). Samples were cut at different angles to the sheet edge (see Figure 12).

Table 1: Mechanical Properties of ILC-AES batch 1 material before (sheet 217) and after (sheet 55) heat treatment for samples cut at different angles to one sheet edge.

BEFORE	Yield strength (MPa)	UTS (MPa)	Elongation (%)	Strain Hard Coeff
0 degree	67	173.2	66.7	0.32
25 degree	63	185.8	60.67	0.35
65 degree	62	185.6	60.04	0.33
90 degree	86	170.1	66.68	0.21
AFTER				
0 degree	46.2	168.5	65	0.38
25 degree	58.1	180.8	59.3	0.36
65 degree	43.2	178.4	60.7	0.41
90 degree	48.4	168.7	65	0.37